63-3-4

DEVELOPMENT OF FLEXIBLE POLYMERS AS THERMAL INSULATION IN SOLID-PROPELLANT ROCKET MOTORS

Quarterly Progress Report
Contract DA-36-034-ORD-3325 RD
January 1 to March 31, 1963

Submitted to:

Rock Island Arsenal
Rock Island, Illinois

April 1963

ATLANTIC RESEARCH
CORPORATION

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April 10, 1963

Commanding Officer
Rock Island Arsenal
Rock Island, Illinois

Attention: ORDBC-9320

Dear Sir:

Enclosed is the Quarterly Progress Report that covers the period from January 1 to March 31, 1963 on Contract DA-36-034-ORD-3325 RD, Flexible Polymer Development.

Very truly yours,

ATLANTIC RESEARCH CORPORATION

T. R. Walton

T. R. Walton
Project Director

cc: Commanding Officer
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ATLANTIC RESEARCH CORPORATION
ALEXANDRIA, VIRGINIA

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April 1963

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ABSTRACT

The mechanical properties of several unfilled and asbestos-filled resins were determined. The elongations of three unfilled epoxy resins varied from 160 to 510 per cent while the elongations of three asbestos-filled epoxy resins varied from 1.9 to 6.3 per cent. An asbestos-filled polyurethane resin had an elongation of 17 per cent. Except for the filled epoxy resin with an elongation of 1.9 per cent, these materials have adequate ductility for service requirements.

Attempts to obtain lower acid numbers for several of the previously prepared polyesters were not too successful because of premature gellation during the condensation. Attempts to prepare two new polyesters were also unsuccessful for the same reason.

Four asbestos-filled moldings were prepared for insulation evaluation from two of the flexible furan resins and two of the flexible polyester resins.

Six motor firings were made this report period in which several asbestos-filled epoxy and polyurethane resins were evaluated. The best performing material was the Epon 828-tetrahydrophthalic anhydride-castor oil formulation filled with 40 per cent of long-fiber asbestos. The char rate of this material in several motor firings varied from 1.5 to 2.7 mil/sec (average of six samples was 2.0). The 40 per cent-castor oil-modified Guardian filled with 40 per cent long fiber asbestos has also continued to perform well. The best performing polyurethane resin gave char rates of 2.5 and 2.3 mil/sec in two different motor firings. All three of these materials show better insulation performance than the best commercially available materials. Initial motor test results indicate the Plastibest 20 offers no advantage over the standard asbestos fiber used as a filler.

I. INTRODUCTION

The purpose of thermal insulation is to prevent excessive heating of the structural parts of a missile by the burning propellant. The insulation must be sufficiently flexible to conform to the shape of the structural parts which strain elastically under high operational pressure. If the insulation does not elongate adequately, it fractures, and the hot propellant gases penetrate to the motor case to cause overheating and failure. When the insulation is bonded to the grain or to the motor case, flexibility is essential to prevent fracture of the bond and possible motor failure by side burning.

The purpose of this project is to develop flexible polymers, which, when combined with appropriate fillers, will be suitable for use as thermal insulation in solid propellant rocket motors. Polymeric systems selected for investigation are epoxies, phenolics, melamines, furans, polyurethanes, and polyesters. The investigation includes the modification of commercially available resins, the synthesis of polymers, and the correlation of polymer structure with performance.

II. RESULTS AND DISCUSSION

A. EPOXY RESINS

1. Mechanical Properties

The mechanical properties of six epoxy resins were determined this report period. Three of these resins were filled with 40 per cent of asbestos fiber. Generally, measurements are made at a strain rate of 4.8 in/in/min to simulate stress conditions during an actual motor firing. However, in the testing of the asbestos-filled samples, this rate of loading exceeded the recording capabilities of the test instrument and a lower strain rate of one in/in/min was used. The results are summarized in Table I. In previous tests, the elongation of the unfilled 40 per cent-castor oil-modified Guardian was 417 per cent.

An insulation with an elongation greater than 5 per cent and tensile strength greater than 500 psi should meet the minimum service requirements for mechanical properties. The last two formulations (5 and 6) in Table I meet these requirements. The Guardian (4) with an elongation of only 2 per cent would be unsuitable. The first three formulations in Table I are unfilled. Although the reduction in elongation caused by asbestos fibers is not always predictable, an unfilled resin with an elongation greater than 50 to 100 per cent and a tensile strength greater than 200 psi should have at least the required minimum mechanical properties when filled with 40 per cent of asbestos. On this assumption, the first two formulations would be acceptable and the third marginal when filled with asbestos.

2. Static-Motor Firing Tests*

Five convergent-section motor firings of filled epoxy resins were completed this report period. The five similar motor firings used a 5600°F aluminized propellant and burned for approximately 60 seconds at high pressure. The results of these motor firings are reported in Tables II through VI. Tested at the same time were twelve samples supplied by the Rock Island Arsenal. These

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* The motor firing tests are part of our Special Projects Office Materials Program for the Department of the Navy, Contract NOw 62-0511.

samples will not be discussed and are reported here only for comparative purposes. It will be pointed out that R. I. A. samples 2, 4, 5, and 7 show excellent insulation performance with relative char rates of 0.56, 0.64, 0.64, and 0.70, respectively (see Table VII). Photographs of the tested samples are shown in Figures 1 through 5.

Identical comparative standards were used for the first four motors (M-316- M-319) to permit a more meaningful comparison between samples in different motors. This comparison is most readily made by determining a relative char rate as reported in Table VII. This relative char rate is calculated by dividing the char rates of the samples by the char rate of the standard tested in the same motor. A material with a relative char rate less than one is better than the standard U.S.R. 3016. The char rate reported for the fifth material (LX1A) in Table VII appears abnormally high. An identical sample directly above this one had a much lower char rate. In addition, the performance of this same material in motor firing M-323 (Table VI) also indicates it to be a much better insulator than suggested in motor firing M-319.

The results reported in Table VII indicate that the 40 per cent castor oil-modified Guardian and the Epon 828-THPA-castor oil formulation are approximately equivalent in their insulation performance and that both are superior to the comparative standard. Previous motor test results and the data in Table VI show the THPA formulation to perform better than the 40 per cent castor oil-modified Guardian.

In the last column of Table VII, the relative char rate was multiplied by the density of the sample. This value represents the weight of insulation needed for a unit time of protection. The lower the char rate × density factor, the better the performance of the insulation. An insulation with a char rate × density factor less then 1.39 is better than the comparative standard. The 40 per cent-castor oil-modified Guardian and the Epon 828-THPA-castor oil formulation are superior to the standard with a value of approximately 1.00 for both, while the Araldite DP437-NMA is inferior with a value of 1.70.

Routinely, a long-fiber asbestos designated 3R100 is used as a filler for our flexible resins. Plastibest 20 is another long-fiber asbestos frequently used by the insulation industry. Both types of asbestos are of the chrysotile variety. In molding LXA, Plastibest 20 was used as a filler instead of 3R100 in the 40 per cent-castor oil-modified Guardian. This molding (LXA) was tested in motor firings M-318 and M-323. Although there was no direct comparison of the performance of the two types of asbestos for the same formulation in the same motor, Table VII indicates little difference, if any, in the performance of the two types of asbestos.

TABLE I

MECHANICAL PROPERTIES OF FILLED AND UNFILLED EPOXY RESINS[a]

Resin Composition	Filler	Number of Samples Tested	Strain Rate in/in/min	Average Ultimate Elongation (%)	Average Ultimate Tensile Strength (psi)	Average Young's Modulus
1. 40% castor oil modified Guardian	Unfilled	2	4.8	380	826	850
2. Oxiron 2000-Empol 1014 (1:1)	Unfilled	4	4.8	160	252	155
3. Araldite DP 437-NMA (1:1)	Unfilled	2	4.8	510	182	280
4. Guardian[b]	40% long-fiber asbestos	4	1	1.9	4297	276,665
5. 30% castor oil modified Guardian[b]	40% long-fiber asbestos	2	1	6.3	604	15,512
6. 40% Epon 828 60% TOMAD[b]	40% long-fiber asbestos	1	1	5.5	2256	67,680

a. All gage lengths were one inch. Test temperature was 83°F.
b. Exact formulations and structures given in Appendix A.

TABLE II

Convergent Section Motor Firing M-316
Results for Filled Resins[a]

Code Number	Formulation[b]	Per Cent Filler	Filler[c]	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
RIA 1	Methyl phenyl vinyl silicone	25 25	Long-fiber asbestos Mineral-filled silicone molding material	--	3.1	--
RIA 2	1 part NBR (45/55) 1 part phenol-furfural resin	32	Long-fiber asbestos	--	1.5[f]	--
RIA 3	1 part NBR (45/55) 0.5 part phenol-furfural resin	39	Long-fiber asbestos	--	2.2[g]	--
XXXXII B	2.8 part Epon 828[d] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (3R100)	1.47	2.2	3.2
ILA	40 per cent-castor oil-modified Guardian 1.0 part Epon 828 1.29 part NMA 1.52 part castor oil	40	Long-fiber asbestos (3R100)	1.47	1.9	2.8
3016	U. S. Rubber Company[e]	50	Asbestos	1.39	2.7	3.8

a. Length of firing: 58.5 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b. Only major components are given for the Rock Island Arsenal samples. The epoxy resins are cured with approximately 2.5 per cent by weight of benzyldimethyl amine.

c. The long-fiber asbestos used in the RIA and ARC samples was Chrysotile, but the source (supplier) may not be the same.

d. The THPA and castor oil were heated together in the presence of 0.7 per cent BDMA for 48 hours at 125°C before mixing with Epon 828.

e. Included in firing as a comparative standard.

f. Uncharred material swelled. Measurements made at edge.

g. Eneven erosion and swelling of uncharred material.

TABLE III

Convergent Section Motor Firing M-317
Results for Filled Resins[a]

Code Number	Formulation[b]	Per Cent Filler	Filler[c]	Density (gm/cc)	Char Rate (mil/sec)	Char Rate x Density
RIA 4	1 part NBR (45/55) 1 part phenol-furfural resin	19	Long-fiber asbestos	--	1.6[f]	--
RIA 5	1 part NBR (45/55) 1 part phenol-furfural resin 0.3 part trioctyl phosphate	17	Long-fiber asbestos	--	1.6	--
RIA 6	1 part SBR (high-viscosity liquid) 0.12 part sulfur	19	Long-fiber asbestos	--	2.5	--
XXXXII B	2.8 part Epon 828[d] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (3R100)	1.47	1.5	2.2
XB	1 part Araldite DP 437 1 part NMA	40	Long-fiber asbestos (3R100)	1.52	2.7	4.1
3016	U. S. Rubber Company[e]	50	Asbestos	1.39	2.5	3.5

a. Length of firing: 55.1 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b-f See Table II.

TABLE IV

Convergent Section Motor Firing M-318
Results for Filled Resins[a]

Code Number	Formulation[b]	Per Cent Filler	Filler[c]	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
RIA 7	NBR-PVC blend	23 12	Long-fiber asbestos Hydrated SiO_2	--	2.3[f]	--
RIA 8	1 part liquid NBR, 0.2 part sulfur	28	Long-fiber asbestos	--	2.5	--
RIA 9	SBR (23.5/76.5)	23 12	Long-fiber asbestos Hydrated SiO_2	--	2.5	--
LXA	40 per cent castor oil modified Guardian 1.0 part Epon 828 1.29 part NMA 1.52 part castor oil	40	Long-fiber asbestos (Plastibest 20)	1.44	2.2	3.2
LXIA	2.8 part Epon 828[d] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (R100)	1.41	2.3	2.2
3016	U. S. Rubber Company[e]	50	Asbestos	1.39	3.3	4.6

a. Length of firing: 60.5 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b-c. See Table II

d. The THPA and castor oil were heated together in the presence of 1.3 per cent BDMA for 29 hours at 150°C before mixing with Epon 828.

e-f. See Table II

TABLE V

Convergent Section Motor Firing M-319
Results for Filled Resins[a]

Code Number	Formulation[b]	Per Cent Filler	Filler[c]	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
RIA 10	1 part SBR (low-viscosity liquid) 0.12 part sulfur	30	Long-fiber asbestos	--	2.6	--
RIA 11	1 part SBR (high-viscosity liquid) 0.12 part sulfur	20 20	Long-fiber asbestos Hydrated SiO_2	--	3.1	--
RIA 12	1 part vinylidene fluoride/hexafluoropropylene 0.5 part polychloroprene	6 15 19	Magnesium oxide Potassium oxalate MT carbon black	--	3.9	--
XXXXIIB	2.8 part Epon 828[d] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (3R100)	1.47	1.5	2.2
LXIA	2.8 part Epon 828[e] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (3R100)	1.41	2.7	3.8
3016	U. S. Rubber Company	50	Asbestos	1.39	2.3	3.2

a. Length of firing: 61.9 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b-d. See Table II

e. The THPA and castor oil were heated together in the presence of 1.3 per cent BDMA for 29 hours at 150°C before mixing with Epon 828.

TABLE VI

Convergent Section Motor Firing, K-323
Results for Filled Resins[a,b]

Code Number	Formulation[c]	Per Cent Filler	Filler	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
V-44	General Tire and Rubber Company[d]	—	Asbestos	1.28	2.8	3.6
V-44	General Tire and Rubber Company[d]	—	Asbestos	1.28	2.9[f]	3.7
LXA	40 per cent-castor oil-modified Guardian 1.0 part Epon 828 1.29 part NMA 1.52 part castor oil	40	Long-fiber asbestos (Plastibest 20)	1.44	2.9	4.2
LXIA	2.8 part Epon 828[e] 3.0 part THPA 4.2 part castor oil	40	Long-fiber asbestos (3R100)	1.41	2.1[g]	3.0

a. Length of firing: 61.4 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b. Two polyurethane resins were tested in this motor and the results are reported in that section in Table XII.

c. The epoxy resins are cured with 2.5 per cent by weight of benzyldimethyl amine.

d. Included in firing as a comparative standard.

e. The THPA and castor oil were heated together in the presence of 1.3 per cent SDMA for 29 hours at 150°C before mixing with Epon 828.

f. Uncharred material swelled. Measurements made at edge.

g. Slight swelling at center.

TABLE VII

Summary of Insulation Performance
of Epoxy Resins from Motor Firings
M-316, M-317, M-318, and M-319

Code Number	Formulation[a]	Density (gm/cc)	Motor Firing	Char Rate[b] (mil/sec)	Relative[c] Char Rate	Relative Char Rate × Density
XXXXIIB	2.8 parts Epon 828 3.0 parts THPA 4.2 parts castor oil	1.47	M-316	2.2	0.81	1.19
XXXXIIB	2.8 parts Epon 828 3.0 parts THPA 4.2 parts castor oil	1.47	M-317	1.5	0.60	0.88
XXXXIIB	2.8 parts Epon 828 3.0 parts THPA 4.2 parts castor oil	1.47	M-319	1.5	0.65	0.96
LXIA	2.8 parts Epon 828[d] 3.0 parts THPA 4.2 parts castor oil	1.41	M-318	2.3	0.70	0.99
LXIA	2.8 parts Epon 828[d] 3.0 parts THPA 4.2 parts castor oil	1.41	M-319	2.7	1.17	1.65
ILA	40%-castor oil-modified Guardian 1.0 part Epon 828 1.29 part NMA 1.52 part castor oil	1.47	M-316	1.9	0.70	1.03
LXA	40%-castor oil-modified Guardian[e] 1.0 part Epon 828 1.29 part NMA 1.52 part castor oil	1.44	M-318	2.2	0.67	0.96
XB	1 part Araldite DP437 1 part NMA	1.52	M-317	2.7	1.08	1.64
3016	U. S. Rubber Company	1.39	--	--	1.00	1.39

a. All formulations were filled with 40% of long-fiber asbestos (3R100) except for number LXA.
b. The char rate for the comparative standard (U.S.R. 3016) tested in each motor is:
M-316 = 2.7, M-317 = 2.5, M-318 = 3.3, M-319 = 2.3.
c. The relative char rate is the ratio of the char rate of the sample to the char rate of the comparative standard test in the same motor. Since the same comparative standard was used in all four motors, the relative char rate can be more favorably compared between the four motors.
d. LXI differs from XXXXII in that it is a new molding
e. In formulation LXA, Plastibest 20 (a long-fiber asbestos) was used in place of 3R100.

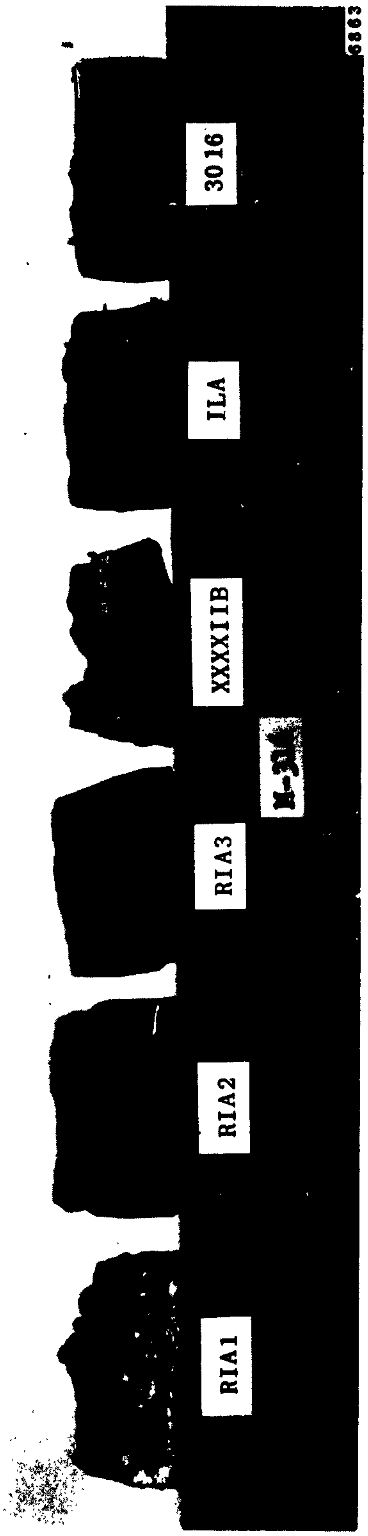


Figure 1: Photograph of Tested Specimens from Motor Firing M-316

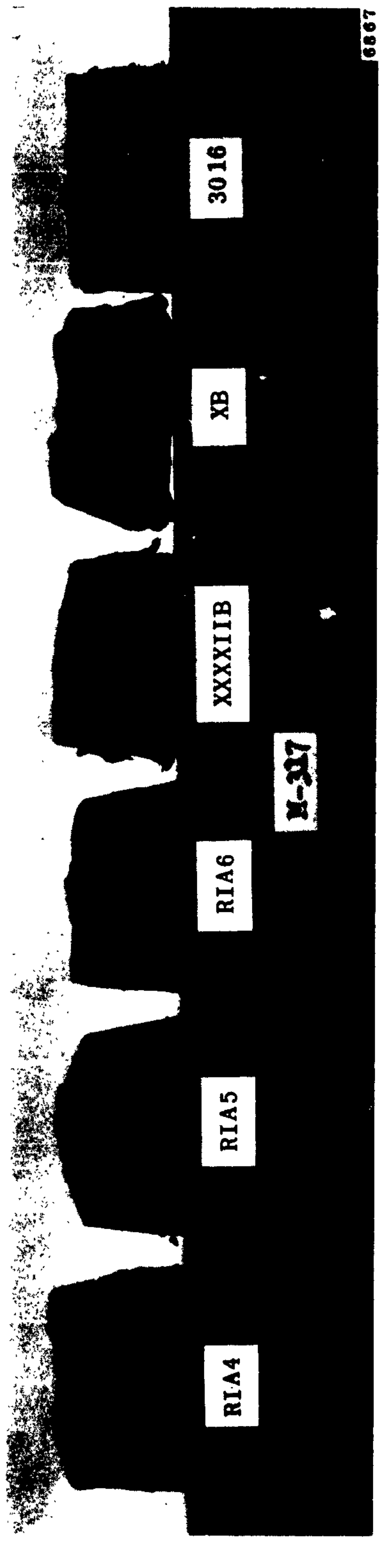


Figure 2. Photograph of Tested Specimens from Motor Firing M-317

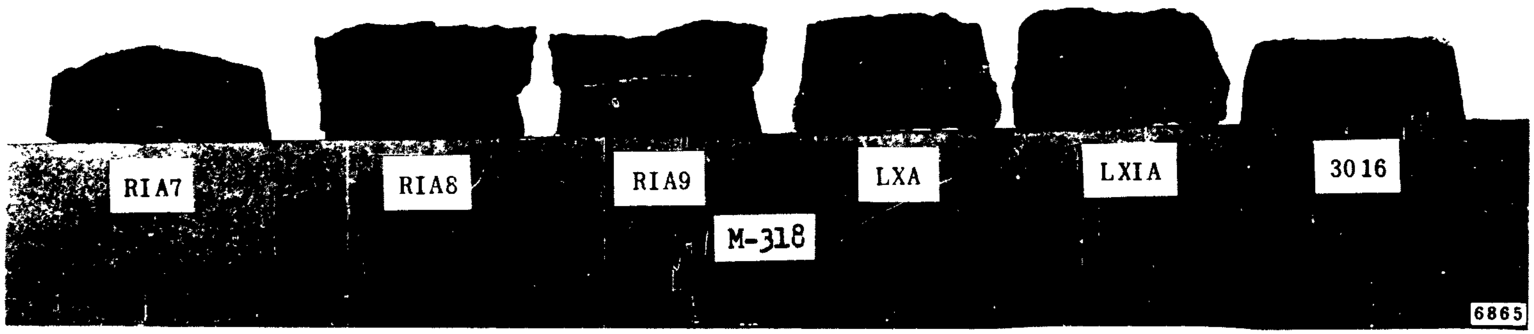


Figure 3. Photograph of Tested Specimens from Motor Firing M-318

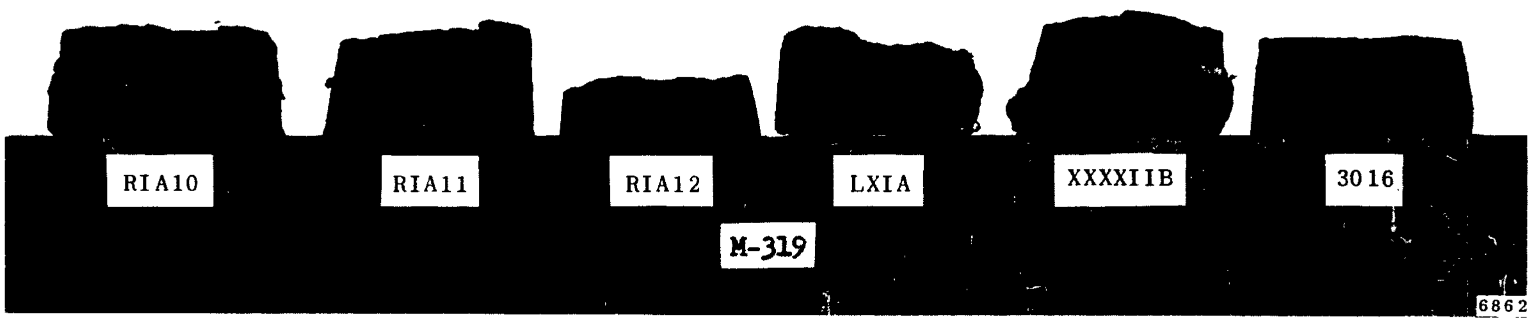


Figure 4. Photograph of Tested Specimens from Motor Firing M-319

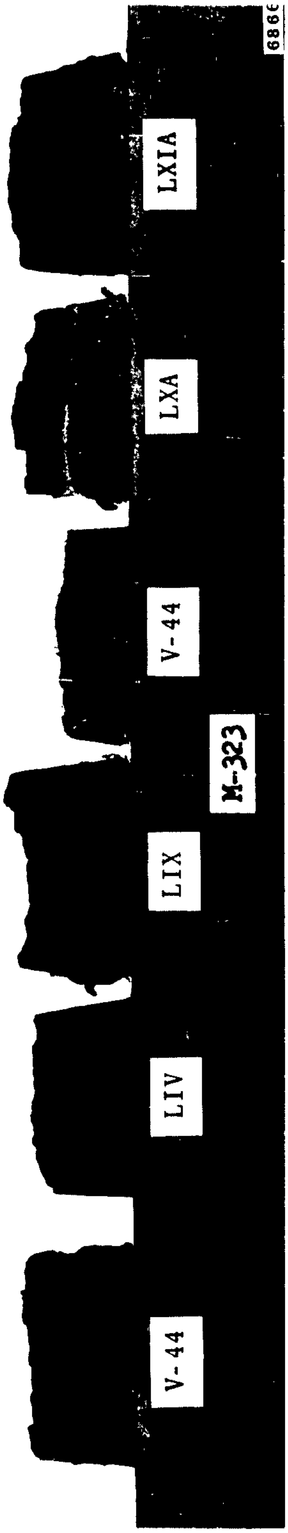


Figure 5: Photograph of Tested Specimens from Motor Firing M-323

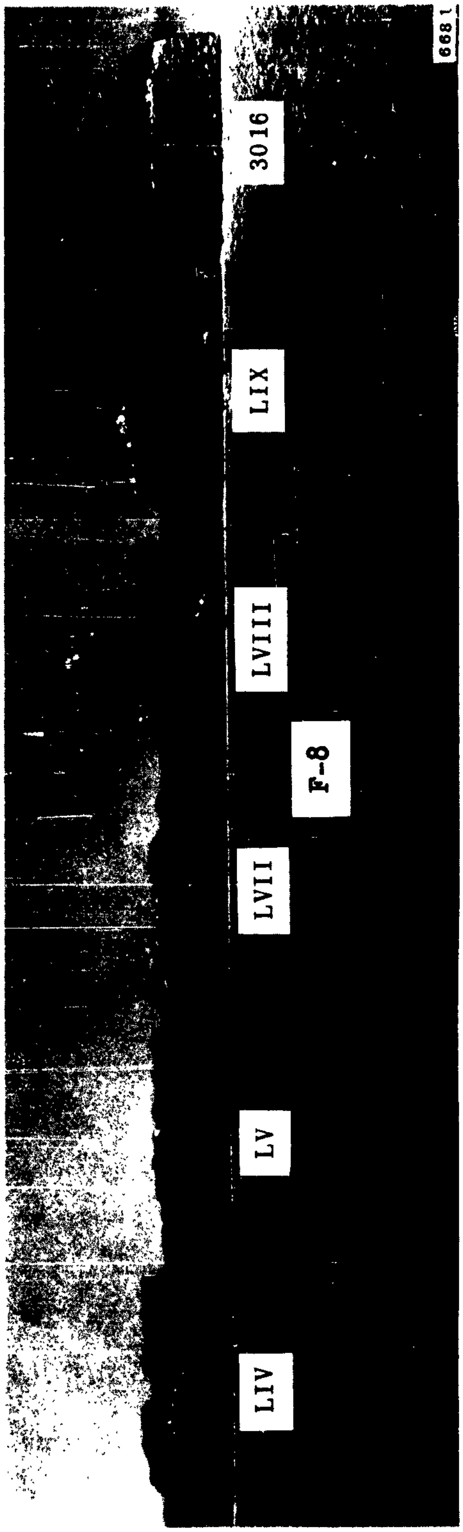


Figure 6: Photograph of Tested Specimensfrom Motor Firing F-8

B. FURAN RESINS

Only two flexible furan resins have been developed which appear to have merit for further evaluation as binders for thermal insulation. The best resin is a furan liquid polymer (Jet Kote X8) - epoxy resin (QZ8-0914) formulation. The cured material (2.5 per cent by weight of BDMA used as a catalyst) is very flexible but splits on bending 90 degrees.

The second resin is an organic acid (dimaleate of 1,4-butanediol) catalyzed cure of a furan liquid polymer (Jet Kote X3M). A low-temperature cure is necessary (50-75°C) or the resin will be rigid. Thus, on heating this flexible resin for 2 hours at 160°C, the material became rigid and brittle. At room temperature, the resin remained quite flexible for 10 weeks with no apparent change. After 6 months at room temperature, the material has become more rigid and brittle, but still has some degree of flexibility. However, because of this poor aging property, it is doubtful whether this latter formulation will be acceptable.

Asbestos-filled loadings have been prepared for both of these formulations and the pertinent information is summarized in Table VIII.

In molding LXIV, an acid-washed asbestos was used. This was prepared by soaking the long-fiber asbestos (3R100) overnight in 5 per cent hydrochloric acid, washing with water until neutral to indicator paper, air drying, and final drying in an 120°C oven. The Diacid 150 was dissolved in the Jet Kote X3M at approximately 50°C and the acid-washed asbestos mixed in a sigma-blade mixer which was cooled with a water jacket. In preparing smaller quantities of asbestos impregnated with this resin, a solvent technique was used. The Jet Kote X3M and Diacid 150 were dissolved in acetone and the asbestos mixed in by hand. The acetone was evaporated under vacuum at 40°C. However, during the evaporation of the acetone, some of the volatile components of the liquid furan polymer (probably furfuryl alcohol or furfural) were also removed. In one case, as much as 25 per cent of the original weight of the resin was lost, and so this technique was not further studied. In molding LXV, the resin and asbestos were mixed in the usual manner with the sigma-blade mixer.

These two moldings will be evaluated in a static-motor firing.

TABLE VIII

Asbestos-Filled Furan Moldings

Code Number	Formulation	Asbestos Content (per cent)	Molding Conditions	Density (gm/cc)
LXIV	9.5 part Jet Kote X-3M[a] 0.5 part Diacid 150[b]	40[c]	75°C/41 hrs/1000 psi	1.63
LXV	1 part Jet Kote X-3M[a] 1 part QZ8-0914 2.5 per cent BDMA	40	120°C/24 hrs/250 psi	1.54

a. Liquid furan polymer
b. Adduct of 1 mole butanediol and 2 moles maleic anhydride
c. Acid-washed asbestos used.

C. POLYESTER RESINS

1. Flexibilization

Most of the polyester work in this report period was oriented toward improving the mechanical properties of the resin. This essentially involved increasing the degree of polymerization of the polyester, that is, carrying the condensation to lower acid numbers. In many of the attempts to obtain lower acid numbers, the polyesters gelled and had to be discarded. The polyester resins studied this report period are summarized in Table IX.

Polyester V was described in the last Quarterly Progress Report (January 1963). It is a viscous liquid which on curing with peroxide yields a very flexible material, but of low tensile strength. The acid number of the uncured polyester was 100. Since the low tensile strength of the cured resin may be due to the low degree of polymerization of the polyester, further condensation of the uncured polyester to a lower acid number was attempted by additional heating at 210°C under vacuum. However, after several hours, the polyester gelled and had to be discarded. The lowest acid number recorded before it gelled was 54.

Since Polyester X had the best mechanical properties of the esters described in the last Quarterly Progress Report (January 1963), a large amount was prepared for molding and insulation studies. This large-scale preparation is designated XB in Table IX. Although the acid number was 15 for the original ester (X), the acid number was 20 in this preparation. The peroxide-cured resin was flexible and split only on repeated flexing of 180 degrees. This polyester was used in the preparation of the asbestos-filled molding LXIII.

A large amount of Polyester II was also prepared for molding and insulation studies. On attempting to reduce the acid number of this preparation below 54, the resin gelled and had to be discarded. In a second preparation, 0.01 per cent hydroquinone was added to the reaction mixture to inhibit gellation. In this preparation, the acid number was 22 (IIC, Table IX). The peroxide cured resin was flexible, but split on bending. This polyester was used in the preparation of asbestos-filled molding LXII.

Attempts were made to prepare two new polyesters this report period. However, on attempting to reduce the acid number of these two polyesters below 63, they gelled even in the presence of 0.01 per cent hydroquinone. Polyester XI was similar to Polyester V (last Q.P.R., January 1963) except that less itaconic acid was used and Polyester XII was similar to Polyester IX except that less maleic anhydride was used. A peroxide cure of Polyester IX with an acid number of 63 resulted in a flexible material, but it was cheesy and broke on bending.

2. Asbestos-Filled Moldings

Asbestos-filled moldings were prepared from polyesters XB and IIC. These two polyesters are highly viscous and a solvent must be used to distribute the resin (and the catalyst) uniformly in the asbestos fibers. Acetone is a convenient solvent to use since it is easily removed under vacuum at temperatures (40-50°C) which do not appreciably cure the resin. With the unfilled polyesters, complete cures are obtained with only 1.1 per cent of dicumyl peroxide as the catalyst. However, with the asbestos-filled loadings, 2.2 per cent of the peroxide catalyst was necessary to obtain a completely cured, tack-free material.

The asbestos-filled moldings of polyesters XB and IIC are described in Table X. Even when filled with 40 per cent of long-fiber asbestos, the 1/4-inch-thick moldings are quite flexible. The 1/2-inch-thick moldings will be evaluated shortly in a static-motor firing.

TABLE IX

Synthesis of Polyester Resins

Code No.	Formulation[a]	Acid No.	Results[b]
V	0.5m butanediol 0.5m propylene glycol 0.45m adipic acid 0.45m itaconic acid	100	Ester gelled on attempting to reduce acid no. below 54.
XB	1m propylene glycol 0.73m succinic anhydride 0.18m maleic anhydride	20	Peroxide-cured resin was flexible and split only on repeated flexing of 180 degrees.[d]
IIC	1m propylene glycol[c] 0.73m adipic acid 0.18m maleic anhydride	22	Peroxide-cured resin flexible, but splits on bending.[d]
XI	0.50m butanediol[c] 0.50m propylene glycol 0.66m adipic acid 0.25m itaconic acid	63	Ester gelled on attempting to reduce acid no. below 63. Peroxide-cured resin was flexible, but cheesy and broke on bending.[d]
XII	1.00m propylene glycol[c] 0.73m sebacic acid 0.18m maleic anhydride	62	Ester gelled on attempting to reduce acid no. below 62.

a. Reaction conditions are described in last Quarterly Progress Report, January 1963.
b. In the peroxide cure , 0.11 per cent dicumyl peroxide was used.
c. 0.01 per cent hydroquinone used as inhibitor.
d. Samples were approximately 60 mm in diameter and 4 mm thick.

TABLE X

Asbestos-Filled Polyester Moldings

Code Number	Formulation[a]	Asbestos Content (per cent)	Molding Conditions	Density (gm/cc)
LXII	Polyester II C 1.0m propylene glycol 0.73m adipic acid 0.18m maleic acid (acid no. = 22)	40	6 hrs/150°C/1000 psi	1.57
LXIII	Polyester XB 1.0m propylene glycol 0.73m succinic anhydride 0.18m maleic anhydride (acid no. = 20)	40	8 hrs/150°C/250 psi	1.55

a. 2.2 per cent of dicumyl peroxide used as catalyst.

D. POLYURETHANE RESINS

1. Mechanical Properties

The mechanical properties of the toluene diisocyanate-castor oil (23/77) formulation containing 40 per cent of long-fiber asbestos (3R100) were determined on four samples at a strain rate of 1 in/in/min. Tests were made at ambient conditions (83°F) and average values of four samples were:

Ultimate elongation (1 inch gage length)	17%
Ultimate tensile strength	1127 psi
Young's modulus	9567 psi

This material would meet the service requirements for mechanical properties. This material was also motor tested and the results are reported in that section.

2. Static Motor Firing Tests

The four asbestos-filled polyurethanes described in the last Quarterly Progress Report (January 1963) and one molded this report period were motor tested and the results are reported in motor firings F-8 (Table XI) and M-323 (Table XII). Photographs of the tested samples are shown in Figures 6 and 5, respectively (page 14). The samples were in the convergent section.

Molding LIX performed better than 3016 in motor firing F-8 and V-44 in motor firing M-323. Molding LIV is comparable to 3016 and V-44 in char rate performance, but its higher density renders it somewhat less effective. Molding LVII did not perform as well as expected. As can be seen from the photograph (Figure 6) of the tested samples, the "F" motor firing (6500°F) is extremely severe on the samples.

TABLE XI

Convergent Section Motor Firing F-8
Results for Filled Resins[a]

Code Number	Formulation	Per Cent Filler	Filler	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
LIV	7.0 parts Adiprene L-100 3.0 parts Epon 812 3.0 parts MOCA	40	Long-fiber asbestos	1.52	3.0	4.6
LV	5.0 parts Adiprene L-100 5.0 parts QZ-8-0914 3.0 parts MOCA	40	Long-fiber asbestos	1.46	4.2	6.1
LVII	2.3 parts toluene diisocyanate 7.7 parts castor oil	40	Long-fiber asbestos	1.41	4.3	6.1
LVIII	10.0 parts Adiprene L-100 0.73 parts Quadrol	40	Long-fiber asbestos	1.35	3.6	4.9
LIX	10.3 parts toluene diisocyanate 10.1 parts hexamethylene diisocyanate 18.0 parts castor oil 13.5 parts polypropylene glycol 150	40	Long-fiber asbestos	1.43	2.5	3 6
3016	U. S. Rubber Company[b]	50	Asbestos	1.39	3.0	4.2

a. Length of firing: 69.1 sec; flame temperature: 6500°F; low pressure; motor quenched with N_2 at end of firing.

b. Included in firing as a comparative standard.

TABLE XII

Convergent Section Motor Firing M-323
Results for Filled Resins[a,b]

Code Number	Formulation	Per Cent Filler	Filler	Density (gm/cc)	Char Rate (mil/sec)	Char Rate × Density
V-44	General Tire and Rubber Company[c]	?	Asbestos	1.28	2.8	3.6
LIV	7.0 parts Adiprene L-100 3.0 parts Epon 812 3.0 parts MOCA	40	Long-fiber asbestos	1.52	2.9	4.4
LIX	10.3 parts toluene diisocyanate 10.1 parts hexamethylene diisocyanate 18.0 parts castor oil 13.5 parts polypropylene glycol 150	40	Long-fiber asbestos	1.43	2.3[d]	3.3
V-44	General Tire and Rubber Company[c]	?	Asbestos	1.28	2.9[e]	3.7

a. Length of firing: 61.4 sec; flame temperature: 5600°F; high pressure; motor quenched with N_2 at end of firing.

b. Two epoxy resins were tested in this motor and the results are reported in that section in Table VI.

c. Included in firing as a comparative standard.

d. Uneven erosion.

e. Uncharred material swelled. Measurements made at edge.

III. SUMMARY OF INSULATION WORK

Numerous flexible epoxy resins have been prepared using external and internal modifiers. Many of these resins have elongations over 100 per cent and some as high as 400 per cent. Asbestos-fiber-filled moldings have been prepared and evaluated for insulation performance in the oxyacetylene torch and in static-motor firings. In addition to asbestos fibers, potassium oxalate, asbestos fiber-potassium oxalate composite, silica microballoons, magnesium carbonate, polyethylene, asbestos fiber-potassium oxalate-silica microballoons, and asbestos powder have been investigated as possible fillers with a few of the more promising flexible epoxy resins. The best-performing resins are summarized in Table XIII.

Flexible phenolic resins have been prepared by modifying A-stage phenolic resins prepared from phenol, or nonyl phenol, and formaldehyde, with long-chain, difunctional compounds such as diepoxides and polysulfides. Elongations have ranged from 17 to 380 per cent. Asbestos fiber and asbestos fiber-potassium oxalate composite moldings have been prepared and evaluated in the oxyacetylene torch and static-motor firings. The more promising materials are reported in Table XIII.

Flexible polyurethane resins have been prepared from toluene diisocyanate and hexamethylene diisocyanate by co-reacting with polyols such as castor oil. A liquid polyurethane (Adiprene) has been cured with several epoxy resins, methylene bis-chloroaniline, and Quadrol to very flexible and tough materials. Several of these flexible polyurethane polymers have been loaded with asbestos fibers and still maintain a high degree of flexibility. These asbestos-filled moldings have been tested in static motors and the most promising are reported in Table XIII.

Considerable effort was devoted to the preparation of flexible melamine resins by attempting to co-polymerize melamine through its amine groups and methylol melamine through its hydroxyl groups, with difunctional compounds such as epoxides, polysulfides, acids, alcohols, isocyanates, etc. Although a few flexible formulations have been developed, asbestos-filled moldings have not been successfully prepared.

Studies with the furan resins have yielded several flexible materials by copolymerizing furan liquid polymers with epoxy resins or diisocyanates and by polymerizing furfuryl alcohol and liquid polymers with a long-chain acid at low temperatures. Two asbestos-filled moldings have been prepared from a furan liquid polymer-epoxy resin formulation and from a furan liquid polymer organic acid formulation.

Polyesters have been prepared from various combinations of aliphatic glycols and dibasic acids (ethylene, propylene, diethylene glycols, butanediol, adipic, itaconic, sebacic acids; maleic, succinic anhydrides). By incorporating carbon-carbon double bonds into the polyesters with unsaturated acids, several of these esters were cross-linked with peroxide to flexible resins. Two of these flexible resins have been loaded with 40 per cent of asbestos fibers for insulation evaluation.

The best insulations developed to date in this program are summarized in Table XIII. The better commercially available motor-case insulations have char rates of approximately 2.7 mil/sec in our test motors. In addition to the insulation performance, the density of the material is also an important consideration in the selection of insulation for missile applications. Other properties being equivalent, the product of char rate and density allows a numerical rating of the material according to over-all effectiveness, with the lowest number representing the best material. The volume requirements of the insulation are neglected.*

It should be emphasized that (1) the data in Table XIII represent only a limited number of tests; (2) char rate differences of 0.5 mil/sec are probably not significant when based on one or two tests; and (3) differences in the propellant used, duration of the motor firing, pressure, and specimen position make it difficult to compare materials tested in different motors. U. S. Rubber Company's 3015 and 3016, and General Tire and Rubber Company's Gen Gard V-44 were used as comparative standards and represent some of the best commercially available insulation materials.

*This point is discussed in the Second Annual Summary Report. This Contract, July 31, 1962, pp. 40, 73.

In summary, the Epon 828-THPA-castor oil formulation, the 40 per cent-castor oil-modified Guardian formulation, and the TDI-HMDI castor oil-polypropylene glycol 150 formulation have good insulation performance based on their char rate and density.

TABLE XIII

Summary of Best Insulation Developed to Date Under This Program

Code Number	Formulation	Filler Content (per cent)	Filler	Density (gm/cc)	Position	Flame Temp (°F)	Time (sec)	Pressure	Char Rate (mil/sec)	Char Rate × Density
XXXXII-B	Epon 828:THPA[a]:castor oil (2.8:3.0:4.2)	40	Asbestos fibers[b]	1.47	Convergent	5600	59.6 55.6 58.5 55.1 61.9	High	1.7(2.5)[c](2.0)[d] 1.8(2.7)[f] 2.2(2.7)[f] 1.5(2.5)[f] 1.5(2.3)[f]	2.5(3.1)[c](2.6)[d] 2.6(3.76)[f] 3.2(3.8)[f] 2.2(3.5)[g] 2.2(3.2)[f]
LXIA	Epon 828:THPA[a]:castor oil (2.8:3.0:4.2)	40	Asbestos fibers[b]	1.41	"	"	60.5 61.9 61.4	High	2.3(3.3)[f] 2.7(2.3)[f] 2.1(2.8)[d](2.9)[d]	3.2(3.2)[f] 3.8(3.2)[f] 3.0(3.6)[d](3.7)[d]
XXII-A	40% castor oil-modified Guardian, varying NMA	20 20	Asbestos fiber[b] pot. oxalate	1.46	"	"	61.6 55.6	High	2.4(2.8)[f] 2.7(2.7)[f]	3.5(3.9)[f] 3.9(3.8)[f]
XXXV-A	40% castor oil-modified Guardian, varying NMA[e]	40	Asbestos fiber[b]	1.46	"	"	61.6	High	2.6(2.8)[f]	3.8(3.9)[f]
XXXV-B	40% castor oil-modified Guardian, varying NMA[e]	40	Asbestos fiber[b]	1.44	"	"	55.6	High	2.9(2.7)[f]	4.2(3.8)[f]
IL-A	40% castor oil-modified Guardian, varying NMA[e]	41	Asbestos fiber[b]	1.47	"	"	61.0 58.5	High	2.8(2.5)[d] 1 9(2.7)[f]	4.1(3.2)[d] 2.8(3.8)[f]
LXA	40% castor oil-modified Guardian, varying NMA[e]	40	Asbestos fiber[g]	1.44	"	"	60.5 61.4	High	2.2(3.3)[f] 2.9(2.8)[d](2.9)[d]	3.2(4.6)[f] 4.2(3.6)[d](3 7)[d]
XIV-F	40% castor oil-modified Guardian, varying NMA[e]	41	Asbestos fiber[b]	1.41	"	6500	43.6	High	3.0(3.2)[c]	4.2(4.0)[c]
X-B	Araldite DP-437:NMA (1.0:1.0)	40	Asbestos fiber[b]	1.52	"	5600	66.1 55.1	Medium High	2.4(2.6)[c] 2.7(2.5)[f]	3.6(3.2)[c] 4.1(3.5)[f]
X-A	Araldite DP-437:NMA (1.0:1.0)	40	Asbestos fiber[b]	1.52	Peripheral	5600	62.3	High	2.4(2.7)[c]	3.6(3.3)[c]
7-19-E	Phenol-formaldehyde stage "A" resin:Syl-Kem 90 (1.0:1.0)	22.5 22.5	Asbestos fiber[b] Pot. oxalate	1.50	Convergent	6500 5600 5600	46.0 68.1 72.7	High High Medium	3.3(3.5)[c] 2.1(3.1)[c] 2.8([illegible].8)[c]	5.0(4.3)[c] 3.1(3.8)[c] 4.2(3.5)[c]
7-16-B	Nonyl phenol-formaldehyde "A" resin:Syl-Kem 90 (3.0:2.0)	45	Asbestos fiber[b]	1.37	"	5600	66.1	Medium	2.7(2.6)[c]	3.7(3.2)[c]
LIX	Toluene diisocyanate-hexamethylene diisocyanate-castor oil-polypropylene glycol 150 (10.3:10.1:18.0:13.3)	40	Asbestos fiber[b]	1.43	"	6500 5600	69.1 61.4	Low High	2.5(3.0)[f] 2.3(2.8)[d](2 9)[d]	3.6(4.2)[f] 3.3(3 6)[d](3.7)[d]

a. Tetrahydrophthalic anhydride.
b. H. K. Porter Company asbestos fiber 3R100.
c. Values for U. S. Rubber Company's 3015 insulation tested at the same time as a comparative standard.
d. Values for General Tire and Rubber Company's Gen-Gard V-44 insulation tested at the same time as a comparative standard.
e. The 40% castor oil-modified Guardian formulation is: Epon 828: Nadic Methyl Anhydride:Castor oil (1.00:1.29:1.52).
f. Values for U. S. Rubber Company's 3016 insulation tested at the same time as a comparative standard.
g. Plastibest 20, Johns-Manville

IV. FUTURE WORK

The final phase of this program will consist of (1) further evaluation of promising materials in motor firings, (2) providing to the Rock Island Arsenal one-pound samples of several of the best flexible resins developed under the contract, and (3) the preparation of the final 3-year summary report.

APPENDIX

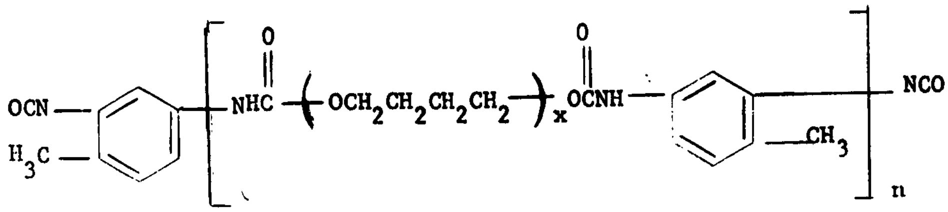
FORMULARY

APPENDIX

FORMULARY

1. Adipic acid (National Aniline): $HOOC(CH_2)_4COOH$

2. Adiprene L (Du Pont):



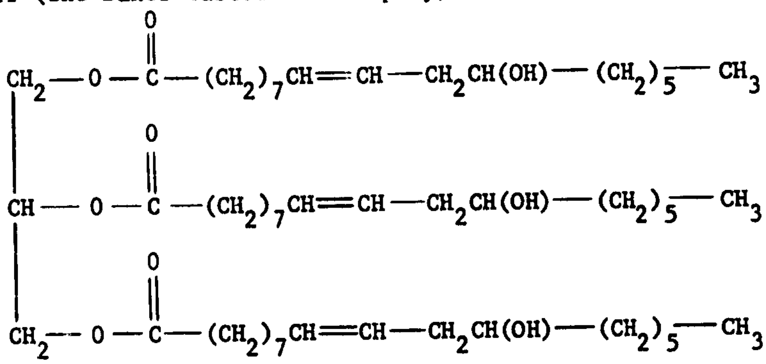
3. Araldite DP-437 (Ciba): an inherently flexible, liquid epoxy resin, epoxy equivalent 285.

4. Asbestos fiber, 3R100 (H. K. Porter Company): chrysotile asbestos fibers.

5. BDMA (Miles Chemical Company): Benzyldimethyl amine

6. Butanediol (Eastman Organic Chemicals): $HO(CH_2)_4OH$

7. Castor oil (The Baker Castor Oil Company):



8. Castor oil-modified Guardian (Atlantic Research Corporation)

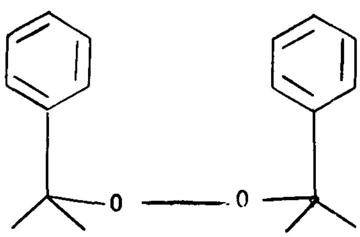
    30% modification
    - 1.00 part Epon 828
    - 1.13 parts NMA
    - 0.91 part castor oil

    40% modification
    - 1.00 part Epon 828
    - 1.29 parts NMA
    - 1.52 parts castor oil

9. Diacid-150

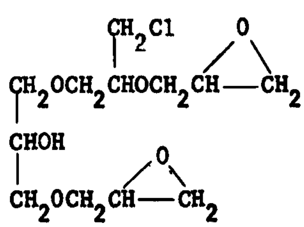
$$HOOCCH{=}CHCOO(CH_2)_4OOCCH{=}CHCOOH$$

10. Dicumyl peroxide (Hercules): R type

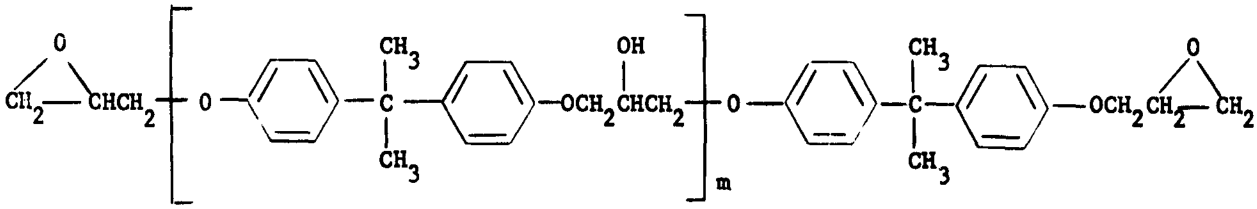


11. Empol 1014 (Emery): a mixture of 95 per cent of C_{36} diacid, 4 per cent of C_{54} triacid, and 1 per cent of a monobasic acid.

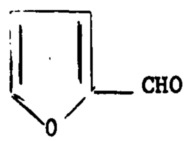
12. Epon 812 (Shell Chemical Corporation): liquid epoxy resin, epoxy equivalent 140-160.



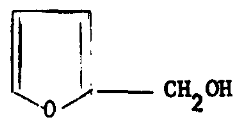
13. Epon 828 (Shell Chemical Corporation): liquid epoxy resin; epoxy equivalent 180-195.



14. Furfural (Fisher Scientific Company):



15. Furfuryl alcohol (Eastman Organic Chemicals):



16. Furfuryl alcohol liquid polymer (Atlas Mineral Products Company): a low viscosity (800-1000 cps) furan polymer.

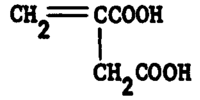
17. Gen-Gard V-44 (The General Tire and Rubber Company): asbestos-filled butadiene-acrylonitrile rubber insulation.

18. Guardian (Atlantic Research Corporation): this material was referred to previously as Standard Guardian. Its formulation is:

10 parts Epon 828
9 parts NMA

19. Hexamethylene diisocyanate (Mobay Chemical Company): $OCN(CH_2)_6NCO$

20. Itaconic acid (Pfizer): methylene succinic acid

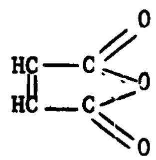


FORMULARY (Cont'd)

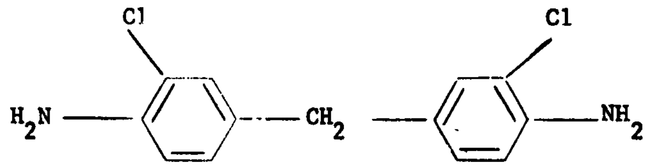
21. Jet-Kote X-3M (Furane Plastics Company): a liquid furfuryl alcohol polymer.

22. Jet-Kote X-8 (Furane Plastics Company): a liquid furfuryl alcohol polymer.

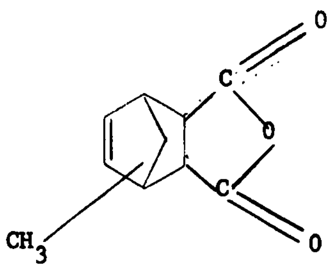
23. Maleic anhydride (Fisher Scientific Company):



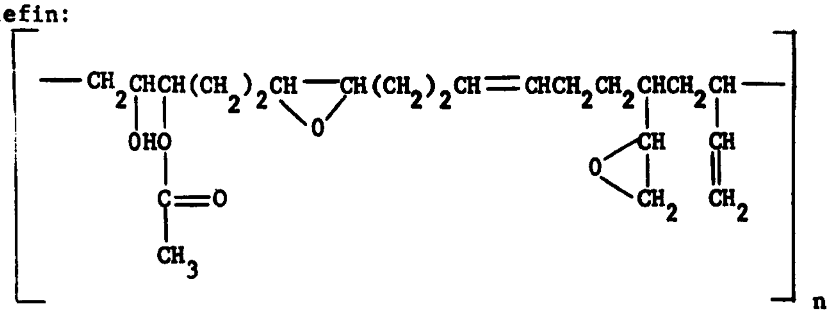
24. MOCA (Du Pont): 4,4''-methylene-bis(2-chloroaniline)



25. NMA, Nadic methyl anhydride (National Aniline):

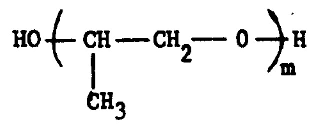


26. Oxiron 2000 (Food Machinery and Chemical Corporation): epoxidized polyolefin:

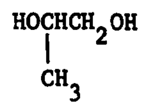


FORMULARY (Cont'd)

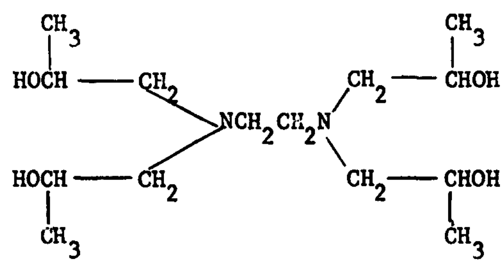
27. Plastibest 20 (Johns-Manville): long fiber asbestos, chrysotile.

28. Polypropylene glycol 150 (Union Carbide): 

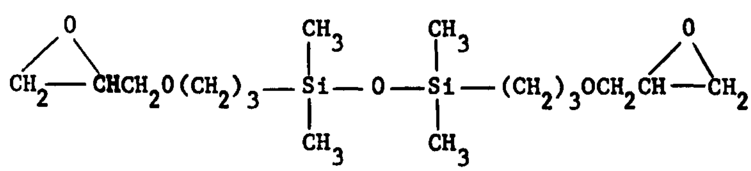
29. Propylene glycol (Union Carbide):



30. Quadrol (Wyandotte Chemical Corporation):

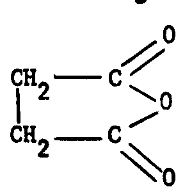


31. QZ-8-0914 (Dow Corning): technical grade.



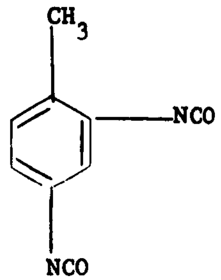
32. Sebacic Acid (Wallace & Tierman): $HOOC(CH_2)_8COOH$

33. Succinic anhydride (Eastman Organic Chemicals)

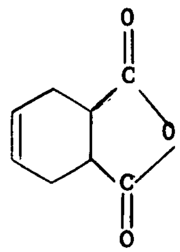


FORMULARY (Cont'd)

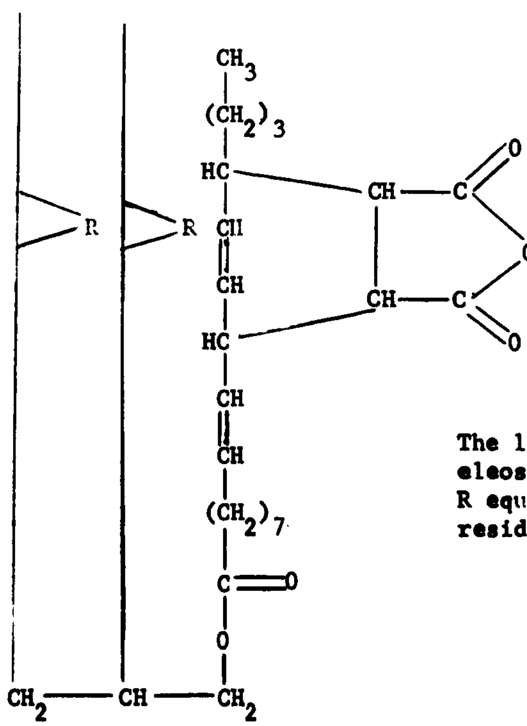
34. TDI (Du Pont, Hylene T): toluene diisocyanate.



35. THPA, tetrahydrophthalic anhydride (Allied Chemical):



36. TOMAD (Atlantic Research Corporation):



The long lines represent the eleostearic acid residue and R equals the maleic anhydride residue.

FORMULARY (Cont'd)

37. U. S. Rubber Company's 3015 insulation (U. S. Rubber): one of the best commercially available insulations. Used as comparative standard in this work. Potassium oxalate filler.

38. U. S. Rubber Company's 3016 insulation (U. S. Rubber): one of the best commercially available insulations. Used as a comparative standard in this work. Asbestos fiber filler.

39. V-44: See Gen-Gard

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Mr. E. J. Wheelahan, ORDXM-RSM (1)
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U. S. Army Munitions Command
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Mr. E. Roffman, ORDBA-1740 (1)
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U. S. Army Materials Research Agency
Watertown Arsenal
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Watertown 72, Mass. (1)

Commanding Officer
Picatinny Arsenal
Attn: Mr. J. J. Scavuzzo, Plastics &
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Mr. D. Stein, ORDBB-DE3
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Picatinny Arsenal
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Commanding Officer
Watertown Arsenal
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Watertown 72, Mass. (3)

Commanding Officer
Watervliet Arsenal
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Watervliet, New York (1)

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Atlantic Research Corporation, Alexandria, Virginia. DEVELOPMENT OF FLEXIBLE POLYMERS AS THERMAL INSULATION IN SOLID-PROPELLANT ROCKET MOTORS. T. R. Walton, E. B. Simmons, and R. O. Thomas. Quarterly Progress Report, April, 1963, (January 1 to March 31, 1963). Contract No. DA-36-034-ORD-3325-RD, Rock Island Arsenal.

Unclassified Report

The preparation of several flexible polyester and furan resins is described. The mechanical properties of 3 unfilled epoxy resins, of 3 asbestos filled epoxy resins, and one asbestos filled polyurethane resin are given. For the asbestos filled resins, the elongation was highest for the polyurethane, 17%. Four asbestos filled moldings were prepared from two of the flexible furan resins and two of the flexible polyester resins. Six static motor firings are reported. The best performing material was the asbestos filled Epon 828-THPA-castor oil formulation with

a char rate as low as 1.5 mil/sec.

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1. Solid propellant motor case insulation.
2. Flexible polymers.
3. Thermal insulations
4. Epoxy resins
5. Furan resins
6. Polyester resins
7. Polyurethane resins
8. Reinforcing materials.
9. Polymer mechanical properties
10. Static motor testing of thermal insulation.

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